SUPERLOW FRICTION OF WS₂ COATINGS IN ULTRAHIGH VACUUM AT LOW TEMPERATURE

Masanori Iwaki
Japan Aerospace Exploration Agency (JAXA), 2-1-1 Sengen, Tsukuba, 305-8505 Japan

Thierry Le Mogne, Julien Fontaine and Jean-Michel Martin
Ecole Centrale de Lyon, LTDS-UMR 5513 CNRS/ECL/ENISE, 36 avenue Guy de Collongue, 69134 Ecully Cedex, France

Shuichiro Watanabe and Junichi Noshiro
Nippon Institute of Technology, 4-1 Gakuendai, Miyashiro-cho, Minami-saitama-gun, 345-8501 Japan

ABSTRACT

Today molybdenum disulfide (MoS₂) is the most commonly used solid lubricant for vacuum applications. Its good tribological performance is considered to be due to its lamellar structure. Coefficient of friction in the millirange (so-called “superlow friction” or “superlubricity”) has already been observed in ultrahigh vacuum at ambient temperature for pure and stoichiometric MoS₂ coatings. On the other hand, tungsten disulfide (WS₂) is also a solid lubricant which has a lamellar structure and works as well as MoS₂ in vacuum. However, it is not widely used nor well studied due to its high cost, and until today there is no report of the achievement of the superlow friction in vacuum. In the present work, we report the superlow friction of WS₂ coatings obtained in ultrahigh vacuum and at low temperature. Pin-on-disk reciprocating friction test for RF sputtered WS₂ coatings on silicon substrates mated against counterpart steel (AISI 52100) pins were conducted in ultrahigh vacuum (3×10⁻⁷Pa) at temperatures ranging from -130 to +200°C. The origin of this superlow friction was discussed.

1. INTRODUCTION

It is known that chemical composition and crystalline structure of MoS₂ films are strongly correlated with their friction properties and wear resistance. Most of vacuum-deposited MoS₂ coatings contain significant amounts of oxygen (10-20 atomic percent) incorporated in their structure, because small amounts of water are present during the sputtering process. The oxygen incorporation affects the crystalline structure and orientation and the film morphology. The effect of increasing the oxygen content in a sputter-deposited MoS₂ film, as studied by XRD and EXAFS is three-fold: (i) an increase in the content of a MoS₃₋ₓOₓ phase (isosstructural with MoS₂ with oxygen substituted to sulphur), (ii) an increase of x in the MoS₃₋ₓOₓ phase and (iii) a decrease of both long-range and short-range orders. A typical shift in the XRD (100) peak is strongly correlated with the oxygen content in the film. This shift has been attributed to the (100) lattice contraction (reduced a₀ lattice constant of about 5% for 15 % oxygen incorporated). It is also correlated with a lattice expansion of the (002) basal planes in the c direction. If the oxygen content has a high value, then molybdenum oxides (MoO_y) would be formed. It has often been claimed that such an expansion in the basal plane distance may result in the lowering of the shear strength (or bonding) between the S-Mo-S sandwiches in the crystallites. Thus, the decreasing of the friction coefficient of MoS₂ coatings in the ultralow regime (friction coefficient of few centigrades) has generally been attributed to the increase of x in the MoS₃₋ₓOₓ phase (i.e. expansion of the basal plane distance). HR-TEM studies show that most of MoS₂ crystallites exhibit many imperfections including faults, kinks and curvature. The origin of MoS₂ curved sheets (or stacks) is not known accurately. It could be attributed to defects preferentially located in one of the S plane of the S-Mo-S sandwiches, causing bending by an asymmetric distribution of bonds around the metal atoms.

In the contrast, pure and stoichiometric MoS₂ film which contains only below 1 % of oxygen can also exhibit extraordinary low friction coefficient. It’s friction coefficient can be in the superlow regime (below 0.01) [1]. The (100) peak of XRD for this MoS₂ film does not shift but agrees well with the data of pure molybdenite crystal. HR-TEM showed the long-range order of the MoS₂ crystallites grown perpendicular to the substrate surface with corresponding electron diffraction pattern. The texture of MoS₂ coatings has been studied by HR-TEM performed on cross-section thin foils. It is generally shown that the first early growth of MoS₂ sheets align parallel to the substrate. However, as the deposition proceeds, the MoS₂
crystals and stacks become progressively aligned perpendicular to the surface.

Even though the ultralow or superlow friction of MoS\textsubscript{2} films in ultrahigh vacuum and it’s mechanism have been studied well as mentioned above, tungsten disulphide (WS\textsubscript{2}), known as a lubricant which has similar properties as those of MoS\textsubscript{2} in ultrahigh vacuum, has not been used nor investigated as much. In this work, we investigate if WS\textsubscript{2} films can exhibit extraordinary low friction as MoS\textsubscript{2} films and how the friction coefficient become in high and low temperatures.

EXPRERIMENTAL

The samples prepared for the experiments were deposited on Si (100) substrates by a RF sputtering deposition process. It’s thickness is 500 nm.

Friction experiments were performed with an analytical ultrahigh vacuum tribometer [2]. The tool consists of a linear reciprocating pin-on-flat tribometer placed inside an ultrahigh vacuum chamber enabled for surface analyses, with X-ray electron spectroscopy (XPS), Auger electron spectroscopy (AES) and an Ar ion gun for surface etching and depth profiling. The pins were all made of AISI 52100 bearing steel with a radius of curvature of 4 mm. Both pin and flat samples were ultrasonically cleaned for 5 min each, first with heptane, then with propanol.

Experiments were conducted in ultrahigh vacuum (10\textsuperscript{-7}Pa) and with a normal load of 3 N, leading to a maximum Hertzian contact pressure of about 470 MPa and a contact diameter of about 110 µm, with a track length of 2 mm and a sliding speed of 0.5 mm/s. And we have conducted them with various temperatures ranging from -130 to 200 °C. Experiments were finished when the number of friction cycles has reached 1000.

RESULTS AND DISCUSSION

Figure 1 shows the average friction coefficient of WS\textsubscript{2} film for various temperatures. A clear tendency was seen: the lower temperature, the lower average friction coefficient. At 130 °C, it has decreased to 0.005, which is in the “superlow friction” regime. For the moment, it is not clear what kind of mechanism works on this superlubricity, but further investigation with surface analysis technique will be one of the solutions for it.

![Figure 1. Average friction coefficient of WS\textsubscript{2} film in function with temperature](image)

REFERENCES
